TOTAL YIELDS FROM HEATED OIL SANDS

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There is at present considerable interest in dry processes for the extraction of bitumen from the Athabasca oil sands. The Lurgi Ruhrgas process is perhaps the best known example of this general technique.

In order to investigate the yield of volatile material from tar sand samples under a variety of conditions, a heated electrical grid apparatus was constructed, basically similar to that used elsewhere[1] for work with coal samples. This permits the heating of small (ca. 20 mg.) samples of oil sand to 900°C at rates up to 10^4 K/sec. The pressure of the surrounding gas is variable between 10 microns Hg and 100 atm. Samples are supported and contained by fine stainless steel mesh, which is heated by a low voltage D.C. circuit. The temperature is measured with a thermocouple in contact with the mesh. Temperature/time profiles are recorded with a high speed chart recorder.

EXPERIMENTAL

Gravimetric measurement of sample weight changes entailed the weighing of the sample with its supporting mesh. Hence it was important to ensure that this did not change in weight on heating. Trial runs in the absence of a sample indicated that hydrogen and helium atmospheres were consistent with this condition, but that carbon dioxide and air were not. The latter exclusion unfortunately precluded gravimetric measurement of the burning of bitumen from the oil sand, which would have provided a valuable comparison with pyrolysis and analytical results.

Further errors in measurements could arise from two phenomena concerning the oil sand itself. The first of these - the oxidation of bitumen by oxygen impurity in the gas, was avoided by the use of ultra high purity gases (hydrogen and helium). The second - "sputtering" of the heated sample through the mesh of its support was eliminated by using stainless steel foil as the support. Total weight loss was not affected by this change, although no solid or liquid material could escape. The use of two folds of mesh was found to contain solids and liquids, while allowing comparatively free transit of gases.

SLOW PYROLYSIS

After the checks described above, a series of heating runs was made on samples of oil sand. A prior analysis of this oil sand, by its supplying laboratory, had indicated 13.8% bitumen, 1.8% water (84.4% sand).

Temperatures up to a maximum of 660°C were reached in this series of runs. One minute was taken to reach the maximum temperature in any given runs, which was held for a further two minutes. The surrounding gas was helium at one atmosphere.

Figure 1 shows the results of 37 such runs. It may be seen that the relationship between temperature and weight loss is approximately linear, though with considerable scatter, up to a temperature of about 500°C, where a maximum is approached. This maximum is about 13%, which may be compared with the nominal total bitumen and water content of 15.6% by analysis.

A straight line which represents a least squares fit to the 27 data points for which T is less than 500°C has been drawn in Figure 1. This is used for comparison with data from other experiments.

EFFECT OF HEATING RATE

Series of experiments were carried out for heating rates of 50 K/sec and 1000 K/sec. These were under the same gas conditions as the slow pyrolysis experiment, and also had the same residence time at the maximum temperature (2 minutes). Figure 2 shows the results of these runs, compared with the straight line fit for Figure 1.

It may be seen that these data are not significantly different from those in Figure 1.

EFFECT OF REDUCED PRESSURE

A number of runs were carried out in helium at lower than atmospheric pressure. Pressures of 400 torr, 200 torr and 7.6 torr were used, without any appreciable effect on the amount of sample volatilized.

EFFECT OF VERY HIGH TEMPERATURES AND OF TIME AT MAXIMUM TEMPERATURE

A small number of runs were carried out in which the samples were rapidly (1000 K/sec.) heated to temperatures well above those used in previous runs.

T_{max}	% Weight Loss
760°C	12.9
910°C	12.8
950°C	12.8

These are all within experimental error of the loss attained at about 500°C.

A series of experiments at 510°C, in which the time the sample spent at 510°C was varied showed that reaction was at least 90% complete at 1 second, and approaching completion by 15 seconds. Heating for several minutes beyond this had no effect.

PYROLYSIS IN HYDROGEN

Pyrolysis of the oil sand in a hydrogen atmosphere was carried out at pressures of 1, 30 and 100 atm. No great difference from the results in helium at 1 atm were noted, except in the case of 100 atm pressure. Here the weight loss of the sample was increased by about 1 - 1-1/2% over the slow pyrolysis case. As elsewhere, the rate of heating appeared to be irrelevant to weight loss.

The quantity of high purity hydrogen required for each run at 100 atm unfortunately precluded extensive investigation of reaction at this pressure.

HELIUM AT HIGH PRESSURE

Helium, at pressures up to 100 atm was also used for a number of runs. In this case, the pressure had a distinct lowering effect on the weight loss of the sample. Again, rate of heating showed no appreciable effect.

Figure 3 shows the data for both hydrogen and helium high pressure work. All runs were to 500°C.

COMPOSITION OF VOLATILIZED MATERIAL

The experimental work described here provided little opportunity to investigate the products of reaction. However, evacuation of the reaction vessel to 100 microns before heating the sample allowed measurement of the gas pressure resulting from reaction. Pressure increases of the order of 100 microns were recorded, but these were consistently higher for slower than for faster heating rates. This implies that the material volatilized has a greater liquid/gas ratio when rapid heating is used.

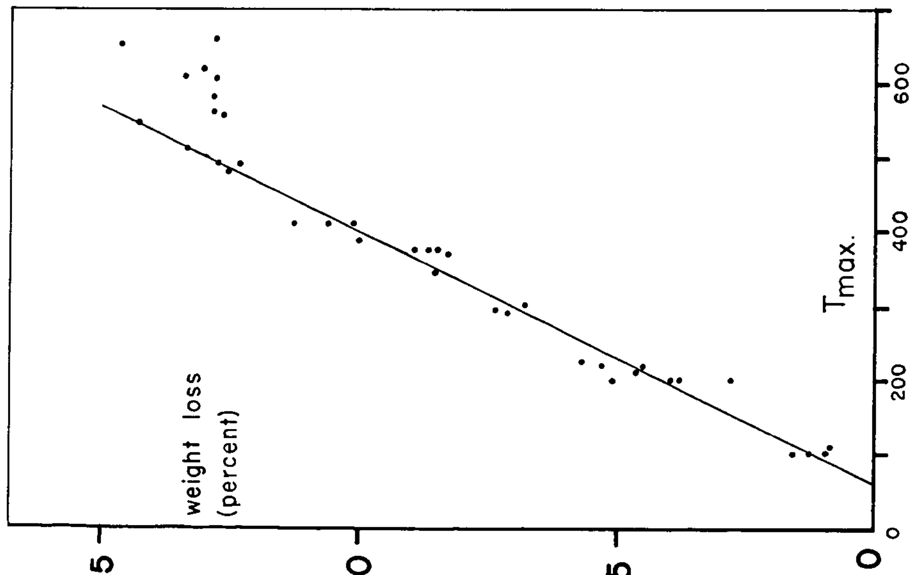
SUMMARY

About 12.9% of the weight of the supplied oil sand is volatilized by heating to 500°C. Rapid heating, higher temperatures, and prolonged heating have little effect on this figure. If the original analysis figures are to be trusted, this implies about 80% yield of bitumen as oil and gas. The oil/gas ratio is probably greater for higher heating rates.

High pressures of inert gas reduce the yield, while high pressure hydrogen increases it. Work in hand on this system includes carbon analyses on the pyrolyzed residues to provide a check on the fate of the bitumen which is not volatilized. A gas chromatographic study of the volatilized material is in the preparatory stages.

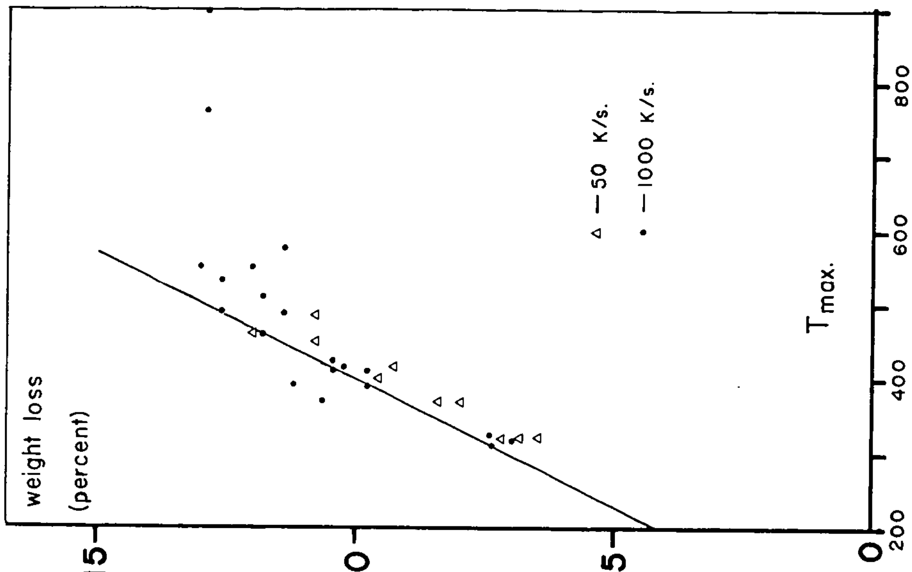
REFERENCE

1. D. B. Anthony, J. B. Howard, H. P. Meissner and H. C. Hottel, Rev. Sci. Instrum. 45 (1974), 992.



weight loss
(percent)
15
10
5
0
0
200
400
600
$T_{max.}$

Fig. 1



weight loss
(percent)
15
10
5
0
200
400
600
800
$T_{max.}$
△ — 50 K/s.
• — 1000 K/s.

Fig. 2

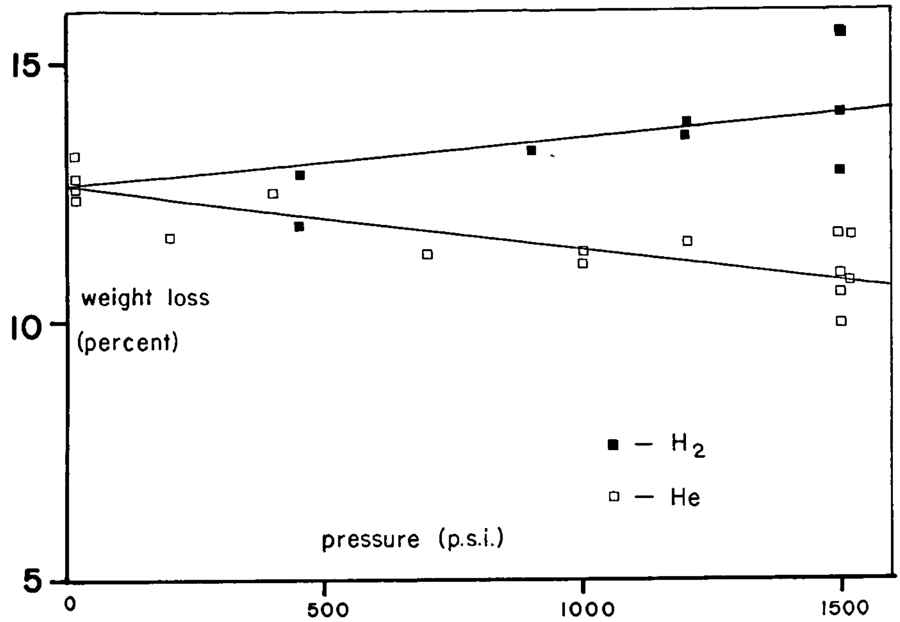


Fig. 3